Work hardening behavior of β Ti-34Nb-2Ta-3Zr-0.5O alloy for orthopedic applications

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ABSTRACT

The β-titanium alloys containing Nb, Ta and Zr, have low elastic modulus and excellent biocompatibility as compared to conventional orthopedic materials like stainless steel, Co-Cr and Ti-6Al-4V alloys. However, one of the drawbacks of these alloys is their low mechanical strength which limits their applicability. Therefore, it is important to improve the strength of these alloys, for which a convenient processing route is cold working. Considering this, a Ti-34Nb-2Ta-3Zr-0.5O (wt.%) alloy has been cold worked to 70% this reduction. This led to 32% increase in the hardness as compared to initial condition. The improvement in hardness was assessed by microstructural investigation which showed presence of micro bands. These bands are mainly responsible for hardening. The formation of these alloys was further investigated by Electron Back Scatter Diffraction (EBSD) analysis. Microbands originated mostly along 112 planes. This microbands were formed due to slip along 112 planes as found in slip trace analysis in EBSD. The mechanical properties were evaluated using micro-Vickers hardness test. So, the present study shows the effect of cold rolling on work hardening behavior of Ti-34Nb-2Ta-3Zr-0.5O at different strain levels.

Keywords : Cold Rolling, Elastic Modulus, Slip Bands.

INTRODUCTION

Biomaterials are naturally-derived materials or hybrids of natural and synthetic origins used in the in vitro application, intended to interact with biological system. Current use of biomaterials in human body includes artificial valves in heart, stents in blood vessels and replacement implants in knees, elbows, hips and orodental structures [1-3]. However, the demand for biomedical implants used for hip, spinal and knee replacements are extremely high amongst all these. A significant fraction of the world population above the age of 60 suffers from degenerative diseases of hip and knee joints, such as arthritis and osteoporesis, and therefore, opt for replacement surgery using prosthetic implants. Artificial biomaterials are the solutions of these problems as surgical implantation of these biomaterials help in restoring the function of the otherwise functionally compromised structures. The materials used for orthopedic implants especially for load bearing applications should possess excellent biocompatibility, high strength, corrosion resistance, low elastic modulus, high wear resistance and not cause toxic reactions inside the body [4].

Mechanical properties – It decides the type of material that can be chosen for specific application. The properties that are of main importance are hardness, tensile strength, modulus and elongation. The response of the material to repeated loading or strains is determined by the fatigue strength of the material. The material replaced for bone is expected to have a modulus equivalent to that of bone. Implants having higher modulus than bone will prevent the necessary stress being transferred to the adjacent bone. It will lead to loss of bone mass (osteopenia) and death of bone cells known as “stress shielding effect”.

Biocompatibility- It is the ability of implants to perform its desired function with respect to medical therapy without eliciting any undesired response [5]. The materials used for implants should not be allergic and not cause toxic reactions in human body. The reaction of the human body to the implant measures the biocompatibility of a material.

High corrosion and wear resistance - The low wear and corrosion resistance of the implants in body results in release of toxic metal ions by the implant into the body. The low wear resistance also results in implant loosening. Thus, development of implants with high corrosion and wear resistance is necessary for the longevity of implant in human system [6].

Osseointegration - Materials with an appropriate surface are essential for the device to integrate well with the adjacent bone when implanted. Surface topography and surface roughness plays an important role in the development of good osseointegration[7].

Currently used metallic biomaterials and their limitations

The materials currently used for implants include 316L stainless steel, cobalt chromium (Co-Cr) alloys and titanium and its alloys. But there are two major problems associated with the first two alloys i.e “Stress Shielding Effect” and Toxicity. Both 316L stainless steel and Co-Cr alloys have higher modulus than bone leading to bone resorption and loosening of implants. Non- compatible ions such as Ni, Cr and Co are found to be released from the stainless steel and cobalt chromium alloys due to corrosion in human body. These ions are associated with long term health problems. Skin related diseases such as dermatitis due to Ni toxicity have been reported and Co is found to be carcinogenic[7]. Ti-6Al-4V has more elastic modulus as compared to bone which has elastic modulus around 4 to 30 Gpa. Cp-Ti has poor mechanical strength as compared to Ti64. β titanium alloys possess low modulus and it possess non toxicity. Certain β-stabilizers such as Nb and Ta have been found to be more effective in reducing the elastic modulus of β-phase [9-10]. However, the beta alloys have some limitations like price is high and the strength is comparatively low in beta solution. Therefore, it requires some hardening to increase the strength. Most common hardening method is aging and work hardening. However, aging treatment gives α and ω phase [3]. The presence of fine precipitates in beta matrix leads to increase in elastic modulus [1]. Other method is work hardening by plastic deformation. Work hardening by cold rolling is one of the common methods for increasing the strength of β titanium alloy[11]. In this study, effect of cold rolling on Ti-34Nb-2Ta-3Zr-0.5O has been studied. Microhardness measurement using Vickers hardness method have been taken. The hardening behavior was studied using microstructural characterization techniques such as Optical Microscopy, Scanning Electron Microscopy and Electron Backscatter Diffraction Method.

EXPERIMENTAL METHOD

Materials and Processing

Ti-34Nb-2Ta-3Zr-0.5O alloy, obtained in the form of pancake, was cast using Tungsten electrode arc furnace by vacuum arc melting. It was hot rolled at 900°C and then solution treated at 900°C for 0.5 h at the same temperature and water quenched. This treatment ensured that the specimen is free from any precipitates and it retains a single-phase BCC type β-microstructure which is generally characteristic of higher temperatures [8]. These samples, are hereafter referred as STQ – Solution-treated & Quenched. The STQ samples were further deformed to 2.5%, 5% and 70% after cold working. Hence the following samples were obtained –1) Solution Treated and Quenched (STQ) 2) 2.5% cold rolled 3) 5% cold rolled and 4) 70% cold rolled.

Microstructural characterization

The microstructures were characterized using optical microscope, SEM (Scanning electron microscope) and Electron Back Scatter Diffraction method. Following standard metallographic techniques specimens were prepared by paper polishing up to 3000 emery paper and then electro polishing in A3 solution at 40V (Struers LectroPol 5). The electropolished samples were etched using Kroll's reagent (2% HF, 6% HNO_3, 92% H_2O) followed by washing with fresh water. The optical and SEM micrographs were recorded with the help of light microscope (Zeiss) and SEM (FEI Helios G10), respectively. Electron backscatter diffraction (EBSD) measurement was performed on 2.5% CR sample from the two alloys in the FEI Helios G10 equipped with an EBSD detector at 25 kV, 5.0 spot size, 13 mm working distance and step size of 1 μm. The samples were prepared in the same way as that of microstructural characterization. The data obtained from EBSD scans were analyzed using commercially available TSL-OIM version 7.0 software (EDAX Inc., Mahwah, NJ).

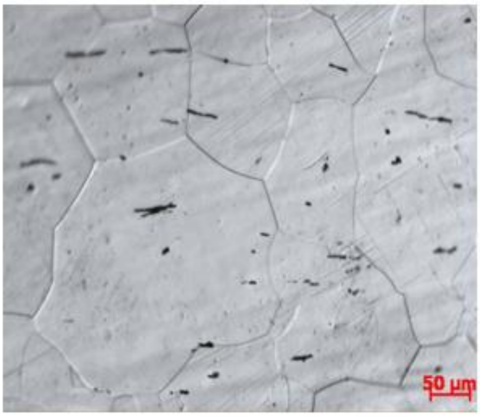
Mechanical characterization

Micro-Vickers hardness were performed to determine the mechanical properties of the specimens. Micro-hardness was measured using 200gf load and 10s dwell time. Atleast 10 readings were taken at randomly selected locations, with center to center distance between two indentations kept at a least a minimum of five times the length of indentation diagonal.

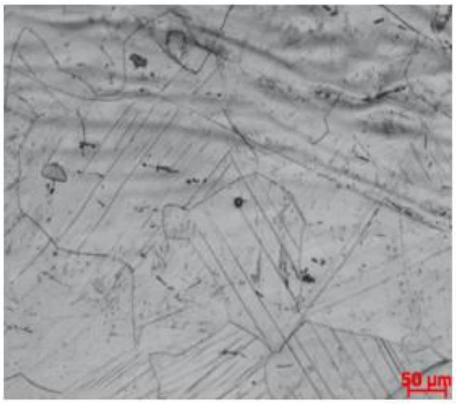
RESULTS & DISCUSSION

Microstructure characterization

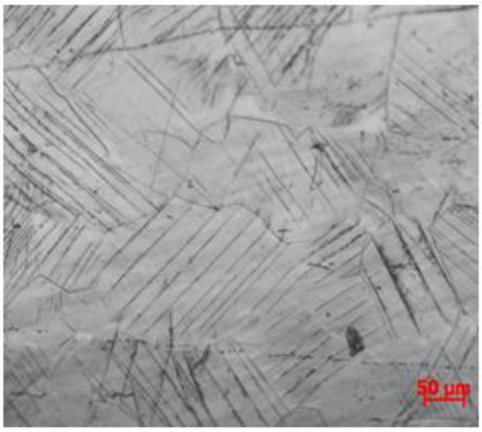
The microstructure of STQ, 2.5% rolled, 5% rolled and 70% rolled samples obtained from optical microscopy clearly reveals the grain boundaries as shown in Fig.1 on the next page. From these figures we can infer that as we increase the deformation on samples, the slip bands start to appear. STQ has equiaxed recrystallized grains. The intensity of slip bands increases with the increase in deformation level. In 70 % cold rolled sample, the slip bands have changed to micro bands due to more strain localization. The SEM images more clearly reveals the slip bands. SEM image 2.5% CR and 5% CR can be seen in Fig.2 (a) and (b) respectively. SEM images have been taken as it would be difficult to visualize the slip bands in optical microscope as deformation level is increased. The EBSD images of 2.5% CR has been shown in Fig.3. Fig.3 (c) shows the Inverse Pole Figure (IPF) analysis of Ti-34Nb-2Ta-3Zr-0.5O sample. After calculating the angle of misorientation in {110} (Fig. 3(a)) and {112} (Fig.3 (b)) slip trace analysis as shown in Table 1, it has been found that misorientation angle is less for {112}. Therefore, slip is more for {112} slip planes.



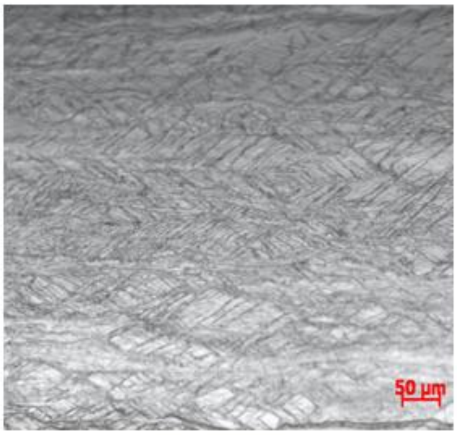
(a)



(b)

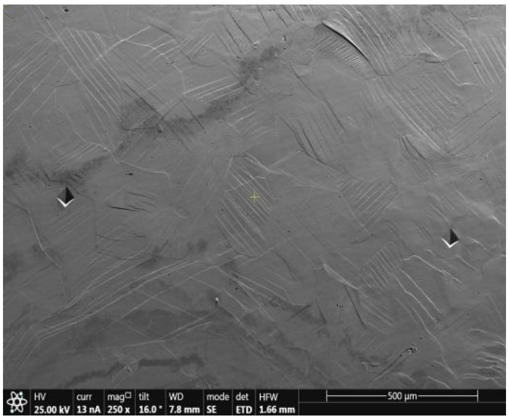


(c)

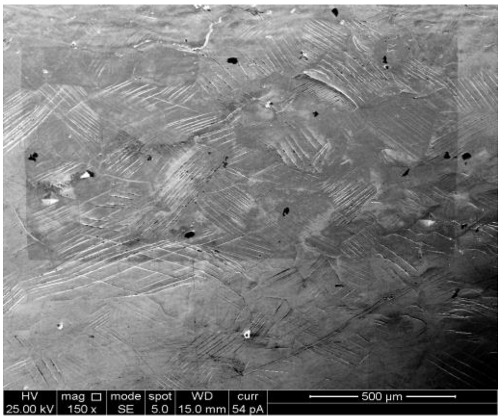


(d)

Fig.1 Optical Micrographs of (a) STQ Sample (b) 2.5 % cold rolled (c) 5% cold rolled (d) 70% cold rolled sample

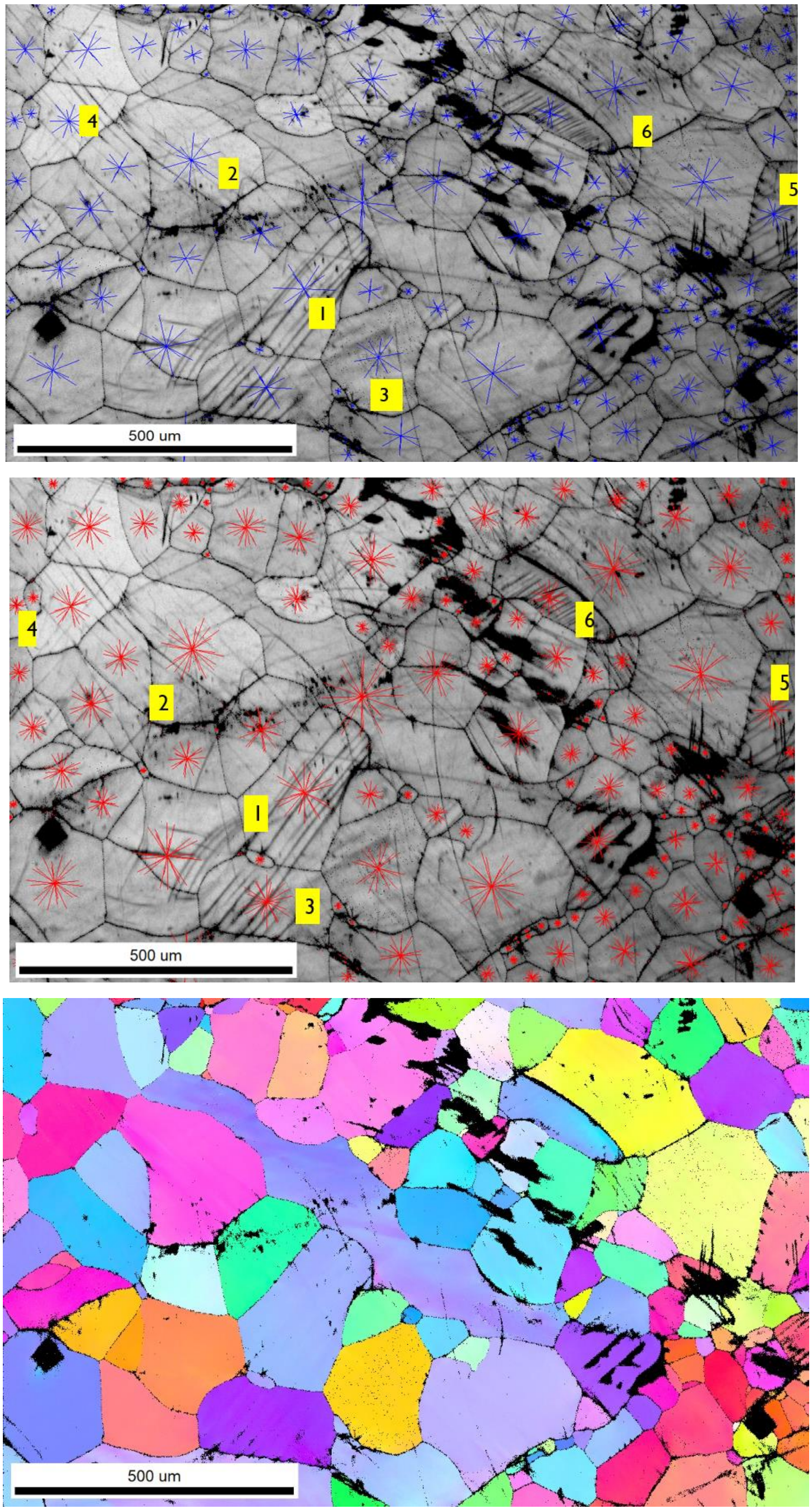


(a)



(b)

Fig.2 SEM images of (a) 2.5% CR and (b) 5%CR



(a)

(b)

(c)

Fig.3 (a) {110} slip traces (b) {112} slip traces (c) IPF maps of 2.5% CR Ti-34Nb-2Ta-3Zr-0.5 O sample

Angle of Misorientation	{110} slip trace	{112} slip trace
Grain 1	14	2
Grain 2	0	3
Grain 3	11	7
Grain 4	0	9
Grain 5	12	1
Grain 6	13	10
Average value	8.33 ± 2.66	5.22 ± 1.71

Table 1: Calculation of angle of misorientation in {110} slip trace and {112} slip trace

Micro Vickers hardness test

The Vickers hardness plot (Fig. 4) compares the micro hardness of the alloys. The plot shows that STQ is having a value of 265.6. 70% cold rolled sample has the maximum hardness of 351.7 and thus reveals that it occurred due to presence of micro bands. 5% rolled sample shows the hardness of 292.6 while 2.5% cold rolled sample has the hardness around 276.31. The change in hardness is due to the increase in the intensity of slip bands. As the percentage of deformation has increased the number of dislocations has increased and it piled up near the slip planes. This created the barrier to the movement of dislocations and hence the material gets work hardened.

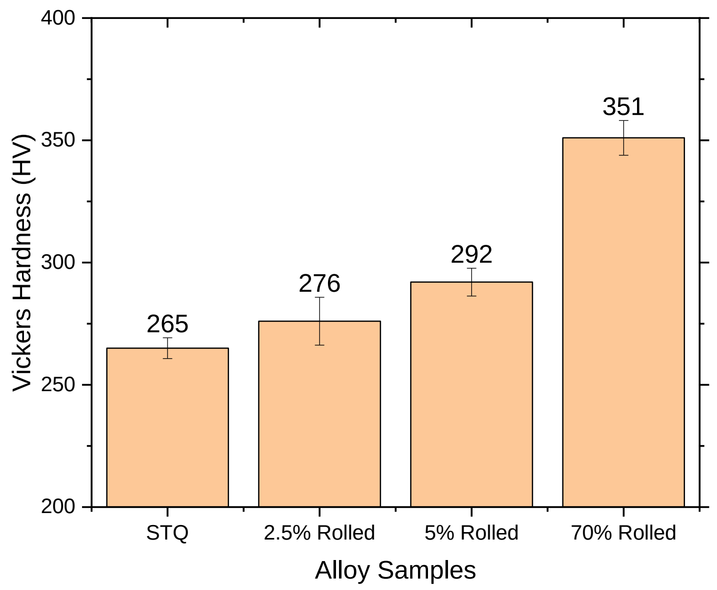


Fig. 4 Micro Vickers Hardness Plot

CONCLUSION

After studying the results and discussion, we obtain that the equiaxed grains were obtained in STQ condition. There were no changes observed in grain shape after 2.5% and 5% cold rolling. However, the grains became elongated after 70% cold rolling. Micro bands were observed in 70% cold rolling. In addition, it has been found that slip bands became more active along {112} planes as confirmed by slip trace analysis of EBSD.

ACKNOWLEDGEMENTS

I would like to thank Dr. Kaushik Chatterjee for giving me opportunity to work on this project. I would also like to thank Srijan Acharya for constantly helping and guiding me as well as giving the technical knowledge of equipment during the project. Finally, I want to express my gratitude to Centre of Bio System Science and Engineering for giving this opportunity to pursue my research interest through BioEngineering Summer Training (BEST) program.

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